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**EFFECTS OF MONOMETHYLHYDRAZINE
ON CRYOPANEL MATERIALS
FOR SPACE SIMULATION CHAMBER
PROPELLION TESTS**

P. G. Waldrep and D. M. Trayer

ARO, Inc.

December 1968

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FOREWORD

The research presented in this report was sponsored by the Arnold Engineering Development Center (AEDC), Air Force Systems Command (AFSC), Arnold Air Force Station, Tennessee, under Program Element 62302F, Project 5730, Task 04.

The results of research were obtained by ARO, Inc. (a subsidiary of Sverdrup & Parcel and Associates, Inc.) contract operator of the AEDC, AFSC, under contract F40600-69-C-0001. The research was conducted from March to August 1967 under ARO Project SW5705. The manuscript was submitted for publication on August 8, 1968.

The authors gratefully acknowledge the assistance of the Metallurgy Department of the Oak Ridge Gaseous Diffusion Plant which is operated for the U. S. Atomic Energy Commission by Union Carbide Corporation, Nuclear Division, in Oak Ridge, Tennessee. This laboratory performed the analyses of the test specimens.

The authors also acknowledge the valuable assistance of S. G. Walker, W. H. Owens, and J. R. McCabe of the Chemical and Metallurgical Branch and H. L. Henderson of the Central Engineering Branch of the Engineering Support Facility, ARO, Inc.

This technical report has been reviewed and is approved.

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ABSTRACT

The effects of monomethylhydrazine on several commonly used cryopanel materials were investigated. The test conditions approximated those expected during rocket engine tests in a space simulation chamber. Monomethylhydrazine gas pressure was 4 torr at room temperature. Test specimen temperatures were varied between 77 and about 300°K. All specimens were tested simultaneously for a total exposure time of 532 hr. Phosphorus deoxidized copper, type 304L stainless steel, and series 1100 aluminum were tested. Some specimens were brazed or welded and some were coated with a black epoxy paint. Several welded and brazed specimens were tested with an imposed bending stress. No microstructural damage occurred to any of the metals. The copper, however, tarnished noticeably, and the cryopanel paint separated from the coated specimens.

CONTENTS

	<u>Page</u>
ABSTRACT	iii
I. INTRODUCTION	1
II. TEST EQUIPMENT	
2.1 Test Chamber	2
2.2 Pumping System	2
2.3 Specimen Holder	3
. 2.4 Monomethylhydrazine Vapor Addition System	3
III. TEST SPECIMENS	
3.1 Brazing and Welding Methods	4
3.2 Stressing Technique.	5
3.3 Copper Specimens	5
3.4 Composite Specimens	5
3.5 Aluminum Specimens	6
3.6 Cryopanel Section.	6
IV. OPERATIONAL PROCEDURE AND EXPOSURE CONDITIONS	
4.1 Operational Procedure	6
4.2 Exposure Conditions	7
V. EVALUATION OF TEST SPECIMENS	7
VI. RESULTS AND DISCUSSION	
6.1 Copper Specimens	8
6.2 Composite Specimens, Copper/304L	9
6.3 Aluminum	9
6.4 Stainless Steel Cryopanel Section.	9
VII. SUMMARY AND CONCLUSIONS	10
REFERENCES	11

APPENDIX

Illustrations

Figure

1. 1.5- by 2-ft Research Vacuum Chamber	15
2. Schematic, MMH Corrosion Test Chamber	16
3. Specimen Holder for MMH Test	17
4. Test Specimens Mounted in Holder.	18

<u>Figure</u>	<u>Page</u>
5. Method of Stressing Test Specimens	19
6. Copper Exposed to MMH, 2X	20
7. Brazed Copper Exposed to MMH, 2X	21
8. Coated Copper Exposed to MMH, 2X	21
9. Coated Brazed Copper Exposed to MMH, 2X	22
10. Coating Stripped from Copper after MMH Exposure, 2X	22
11. Copper Control, Unexposed, 500X.	23
12. Uncoated Copper Exposed to MMH, 500X.	23
13. Coated Copper Exposed to MMH, 500X.	24
14. Brazed Copper Exposed to MMH, Stressed, and Uncoated, 500X	24
15. Control Specimen, Brazed Copper (at Weld), Unexposed, and Unstressed, 500X.	25
16. Brazed Copper Exposed to MMH, Stressed (at Weld), 500X	25
17. Composite Exposed to MMH, Uncoated, 2X.	26
18. Composite Coated and Exposed to MMH, 2X	26
19. Composite, Stainless Steel Side of Weld, Stressed and Exposed to MMH, 500X.	27
20. Control Composite, Stainless Steel Side of Weld, Unexposed, 500X	27
21. Composite, Stainless Steel Portion away from Weld, Stressed and Exposed to MMH, 500X.	28
22. Control Composite, Unexposed, Stainless Steel Portion, 500X.	28
23. Composite, Copper Side of Weld, Stressed and Exposed to MMH	29
24. Control Composite, Copper Side of Weld, Unexposed, 500X	29
25. Composite, Copper Portion away from Weld, Stressed and Exposed to MMH, 500X	30

<u>Figure</u>	<u>Page</u>
26. Aluminum Exposed to MMH, 2X	30
27. Aluminum Exposed to MMH, 500X	31
28. Control Aluminum, Unexposed, 500X	31
29. Stainless Steel Cryopanel Exposed to MMH, 500X	32
30. Control, Stainless Steel Cryopanel, Unexposed, 500X	32

SECTION I INTRODUCTION

Rocket engines using monomethylhydrazine (MMH)* fuel have been tested in space simulation chambers under very high altitude conditions. Chambers for such tests rely on cryogenic pumping to maintain altitude pressures during firing sequences. The cryopanels are thus exposed not only to rocket exhaust products but to raw fuel and oxidizers from propellant leads, tails, and leaks. In addition, inadvertent propellant spills may subject the cryopanels to relatively large amounts of propellant vapors and cryodeposits. Since MMH is a very reactive chemical it could cause substantial damage to cryopanel surfaces.

The reactivity of MMH generally lies between that of more active hydrazine and less active unsymmetrical dimethylhydrazine (UDMH). The material compatibilities of hydrazine and UDMH have been investigated extensively, and much work has been done in understanding the effects of MMH on materials of construction (Refs. 1 through 4). While this information is useful in designing storage, transfer, and propulsion systems, it is not necessarily applicable to the design of space chamber cryopanels since the conditions of exposure are widely different. The cryopanel surfaces operate in high vacuum and experience temperature variations from cryogenic to ambient and perhaps higher. The effects of variations in pressure and temperature on the chemical and physical nature of the propellant and the cryopanel surface may be significant to the ultimate performance of the cryopanel in the simulated space environment. Corrosion rates may be very different from those occurring under conditions of relatively constant pressure and temperature.

The purpose of this study was to make a preliminary investigation into the corrosion behavior of several basic cryopanel materials under conditions that would approximate those inside a space chamber during rocket engine tests in which MMH fuel was used. The room temperature MMH pressure of 4 torr was selected to represent an extreme situation in which raw fuel might be dumped inside a test chamber. As the test

*Propellant grade MMH which conformed to the analysis of Military Specification MIL-P-27404-MMH was used for these tests.

materials were cooled to liquid-nitrogen (LN_2) temperatures and non-condensable gases were pumped off, the chamber pressure dropped to the 10^{-7} torr range. The temperatures of the test materials were cycled between 77 and about 300°K periodically throughout the test period.

The materials chosen for testing were type 304L stainless steel, phosphorus deoxidized copper, and series 1100 aluminum. These metals are commonly used as cryopanel materials. All specimens were tested simultaneously in variations of welding, with protective coatings, and with applied bending stress. The test specimens are described in detail in Section III.

SECTION II TEST EQUIPMENT

2.1 TEST CHAMBER

The 1.5- by 2-ft Research Vacuum Chamber was used. The chamber is pictured in Fig. 1, Appendix I, and shown schematically as it was used for this test in Fig. 2. The chamber is a stainless steel cylinder, 1.5 ft in diameter by 2 ft high and has an operational free volume of 103 liters. A port is provided in the top for introduction of the test specimen holder, and a viewing port is located on the side. The primary chamber components are vacuum sealed with Buna-N® O-rings.

2.2 PUMPING SYSTEM

The chamber is fitted with an LN_2 -trapped mechanical roughing pump, an ion getter pump, and three LN_2 -cooled cryopumps. The ion-getter pump is rated at 840 liters of air per second at 10^{-7} torr. The chamber can be pumped below 10^{-6} torr with this pump. The three LN_2 cryopumps were used in sequence to transfer MMH to the test specimens. The first cryopump, a small stainless steel cylinder 2 in. in diameter by 8 in. long, was used for the initial condensation of the MMH vapor. The second cryopump consists of a stainless steel cylindrical shroud surrounding the specimen holder but having no thermal contact with it; it is open at the bottom and top. The corrodent was deposited onto this shroud as a second step in the transfer to the specimens. The third cryopump consists of the specimens and specimen holder.

Chamber pressures were measured in the vacuum region with an ion gage and a thermocouple gage. A variable reluctance differential pressure transducer was used to measure the pressure of corrodent gas. The transducer was referenced to the mechanical pump vacuum and was equipped with a chart recorder readout.

An LN₂-cooled MMH trap is installed between the chamber and the mechanical pump cold trap. This was used to collect MMH vapors during chamber pumpout.

2.3 SPECIMEN HOLDER

The specimen holder, Fig. 3, consists of a stainless steel cylinder 1.25 in. in diameter by 1 ft long to which four longitudinal metal fins were welded. The test specimens were bolted to these fins. Three of the fins were copper and one was aluminum. Test specimens were mounted by bolting one end between a fin and a metal clamping strip which was cut and drilled to match the fin (Fig. 4). Liquid nitrogen circulated through the central cylinder during cooling cycles. Stainless steel sheathed thermocouples were attached to the base of each fin for temperature measurements.

The holder was inserted through the top of the chamber and vacuum sealed with Buna-N O-rings. The holder was situated inside the LN₂-cooled shroud during testing.

2.4 MONOMETHYLHYDRAZINE VAPOR ADDITION SYSTEM

Liquid MMH was stored in a type 304L stainless steel vessel under its own vapor pressure. Vapors were taken from over the liquid and valved into the chamber by stainless steel needle valves having fluorocarbon packings and seats and Buna-N O-rings. The transfer lines were made from 0.25-in. stainless steel tubing. During introduction of MMH vapor the pressure was monitored by the pressure transducer.

SECTION III TEST SPECIMENS

With the exception of one section of commercially available stainless steel cryopanel, all test specimens were 1 by 2 in., cut from 0.020-in.-thick shim stock. One half of each specimen was covered during testing by the holder fin and the clamping strip. Neglecting thickness, this left 2 in.² of specimen surface available for exposure to the test gas.

Altogether, 36 shim specimens were exposed. All of the shim specimens directly contacted copper holder fins during the test except six of the plain aluminum specimens which contacted the aluminum fin.

The types of specimens tested were:

1. Plain copper
2. Brazed copper
3. Copper coated with black epoxy paint
4. Brazed copper coated with black epoxy paint
5. Brazed copper, stressed
6. Copper/304L stainless steel composites joined with commercially available nickel welding wire
7. Copper/stainless composites coated with black epoxy paint
8. Copper/stainless composites, stressed
9. Plain aluminum
10. One stainless steel 304L cryopanel section

Control specimens of each type were prepared so that the effects of the test could be compared with the unexposed materials. The control specimens were cleaned by the same procedures followed for test specimens, but they were not subjected to applied bending stress and were not exposed to the test environment.

3.1 BRAZING AND WELDING METHODS

Braze joints and welds were made by joining two pieces along a line parallel to the holder fin. The weld bead was halfway between the edge of the fin and the tip of the specimen. Prior to joining, the two pieces were matched and clamped into a special jig. Argon gas was fed through the jig and shielded the underside of the weld against excessive oxidation. Both welding and brazing were done with a DC welder using argon shielding. Welding wire of 0.030-in. diameter was hand fed during joining.

The brazing wire used to join copper specimens was phosphor bronze C having a composition of 95-percent bronze and 5-percent tin.

The copper/stainless steel composite specimens, comprised of a stainless steel 304L tip and a phosphorus deoxidized copper base, were joined with nickel welding wire.

3.2 STRESSING TECHNIQUE

Ten specimens were tested with an imposed external bending stress of just under the yield strength at the edges of the welds. This was done as shown in Fig. 5. Specimens were stressed in matching pairs. A pair of specimens were clamped into the holder at the same point with a metal spacer 0.10 in. thick separating their clamped bases (Fig. 5a). The tips were then separated until the calculated stress level at the edges of the weld was near yield strength (Fig. 5b). Finally, a Teflon® wedge was inserted between the tips to ensure this stress level would be maintained (Fig. 5c).

3.3 COPPER SPECIMENS

Seventeen copper specimens were tested. Nine of these were brazed with phosphor bronze, and four of the brazed specimens were stressed during the test. Three of the unbrazed specimens were coated with black epoxy paint. Two of the unstressed, brazed specimens were coated. All copper specimens contacted copper holder fins during exposure.

The copper specimens were cleaned by (1) polishing with a commercial metal polish, (2) rinsing in technical grade methanol and reagent grade acetone, (3) rinsing in reagent grade methanol, and finally, (4) by ultrasonic cleaning in Freon MF®. They were stored in a vacuum desiccator prior to mounting.

3.4 COMPOSITE SPECIMENS

These 2- by 1-in. specimens consisted of a piece of phosphorus deoxidized copper 1.5 in. long by 1 in. wide welded with nickel welding wire to a piece of 304L stainless steel 0.5 in. long by 1 in. wide. One inch of the length of the copper portion was inserted in the specimen holder, leaving 1 in.² of each material surface exposed to the corrodent.

Eleven composite specimens were exposed. Six were stressed, and two of the unstressed were coated with black epoxy paint.

The stainless steel and weldment portions were cleaned by wire brushing followed by multiple rinses in methanol and acetone and finally by ultrasonic cleaning and degreasing in Freon MF. The copper portion was protected during brushing with masking tape. The copper portions were cleaned by the method described for copper in Section 3.3.

3.5 ALUMINUM SPECIMENS

Eight aluminum specimens were tested with two contacting a copper fin and six contacting an aluminum fin. None was stressed and none was coated.

These specimens were cleaned by washing in multiple baths of methanol and acetone and finally by ultrasonic cleaning and degreasing in Freon MF.

3.6 CRYOPANEL SECTION

A specimen was prepared by cutting a 5.25-in.-long by 1.75-in.-wide section from a commercially available 304L stainless steel cryopanel, fusion welding the edges, and attaching it with welded 0.375-in. stainless steel lines directly into the central LN₂ reservoir of the holder. With this arrangement LN₂ could circulate directly through the specimen during cooling cycles. A sheathed thermocouple was attached to the surface.

This specimen was cleaned by wire brushing the weld areas and washing with methanol and acetone. Finally, the entire specimen holder, with cryopanel specimen, was ultrasonically cleaned and degreased in Freon MF.

SECTION IV OPERATIONAL PROCEDURE AND EXPOSURE CONDITIONS

4.1 OPERATIONAL PROCEDURE

The primary chamber components were disassembled and cleaned prior to the test by scrubbing with an aqueous detergent solution and rinsing with deionized water followed by rinsing with technical grade isopropanol. The system was air dried and pumped to 10⁻⁶ torr before the specimens were inserted.

After the specimen holder was inserted into the test position, the chamber was again pumped to 10⁻⁶ torr and the ion-getter pump isolated. Monomethylhydrazine vapor was then admitted to a pressure of 4 torr as indicated by the pressure transducer readout.

When the specimens had been exposed to the vapor for periods ranging from 8 to 72 hr, they were taken through a temperature cycle. The small cryopump was first cooled with LN₂ to condense the MMH; the chamber pressure typically dropped to 0.1 torr when the cryopump was cooled. Next, the shroud surrounding the specimen holder was cooled with LN₂, and the noncondensable gases were pumped off with the mechanical pump. The small cryopump was then defrosted with a warm nitrogen gas purge and the MMH transferred to the shroud.

The specimen holder was then cooled to 77°K and the shroud warmed by a heated nitrogen purge. The MMH transferred to the specimens by this process was kept there for periods between 3 and 8 hr. Finally, the specimen holder was warmed, completing the temperature cycle, and the chamber pumped out in preparation for the next exposure sequence. The specimens were taken through 12 such cycles with fresh charges of MMH vapor introduced before each sequence.

The pumpdown time increased with subsequent cycles because of contamination of chamber components and test specimens with MMH and its decomposition products. The epoxy paint on the coated specimens absorbed MMH as evidenced by the blistering and separation of the coatings (Section VI) and by the definite odor of MMH on the coated specimens after the test.

4.2 EXPOSURE CONDITIONS

All specimens were tested together in one test so that exposure conditions were the same for every specimen. The specimens were exposed for a total of 458 hr to MMH vapor at temperatures between room temperature and the temperature at which the vapor began to deposit on the cryopumps. The specimens were exposed to MMH cryodeposit for a total of 67 hr. Roughly, 7 hr of test time were spent between these conditions when the specimens were warming and were in contact with both cryodeposit and vapor. Thus, the total exposure time through the 12 temperature cycles was 532 hr.

SECTION V EVALUATION OF TEST SPECIMENS

The specimens were investigated for evidences of corrosion mainly by visual microscopic examination of polished cross sections at high optical magnifications (up to 1000X). Unexposed control specimens were

similarly examined for comparison. Photomicrographs of typical metallographic sections and critical areas were made at 500X. The specimens were prepared for metallographic examination by conventional mounting, polishing, and etching techniques. Particular attention was given to the areas in the vicinity of welds and bends for evidences of stress-corrosion cracking. Grain boundaries and surfaces were closely examined for intergranular corrosion and pitting.

Surface films on the exposed specimens were examined by electron microprobe analysis and by X-ray diffraction in an attempt to identify their chemical compositions.

SECTION VI RESULTS AND DISCUSSION

Analyses of surface films by electron microprobe and X-ray diffraction were largely inconclusive. Carbon was identified in the film by the microprobe, but no association of the carbon with definite chemical compounds was possible by X-ray diffraction analysis.

6.1 COPPER SPECIMENS

All uncoated copper specimens, including the brazed ones, were tarnished from the MMH exposure, Figs. 6 and 7. The tarnishing was not uniform but had a pattern suggesting that it was formed during melting and runoff of the MMH deposited on the surface. The tarnish film was soft and easily removed from the specimens by rubbing. Microprobe analysis of the film along the lower edge of a specimen indicated the presence of carbon, leading to the belief that the deposit consisted of MMH decomposition product.

The black epoxy coating had separated from the copper surfaces, Figs. 8 and 9, and was easily removed. Apparently, the decomposition of absorbed MMH into volatile substances at the copper surface resulted in fracture of the metal-to-coating bonds, followed by blistering during subsequent pumpdown. The metal surface under the coating, Fig. 10, showed tarnishing similar to that observed on the unprotected copper surfaces.

Metallographic examination of the copper specimens revealed no evidence of pitting, intergranular corrosion, or stress-corrosion cracking. A photomicrograph of an unexposed copper control specimen is shown in Fig. 11. Figures 12 and 13 show test specimens tested without black epoxy

coating and with the coating, respectively. No corrosive attack is evident in either case. Brazed and stressed specimens showed no evidence of corrosive activity as can be seen in Fig. 14. Weldments were likewise unaffected in a structural sense by the exposure. Cross-sectional photomicrographs of a control and a stressed test specimen at the welds are shown for comparison in Figs. 15 and 16.

6.2 COMPOSITE SPECIMENS, COPPER/304L

The copper portions of the composites were tarnished in the same manner as the copper specimens discussed in Section 6.1. A typical, uncoated specimen is shown in Fig. 17. The stainless steel portions showed only discoloration caused by welding.

The black epoxy coating had separated from the copper portion of the coated specimens, Fig. 18. Although the coating at first appeared to be well bonded to the stainless steel portion, close microscopic examination of cross sections indicated that it was separated from a large portion of the steel surface. There was no evidence that the coating on the steel had blistered as it had on copper.

Neither the copper, the stainless steel, nor the nickel weldments showed evidence of corrosive attack on the metal itself. No pitting or intergranular corrosion was discovered. The stress imposed on several specimens did not result in detectable stress-corrosion. Photomicrographs of test and control specimens are shown in Figs. 19 through 25. A control specimen for comparison with the copper portion shown in Fig. 25 may be seen in Fig. 11.

6.3 ALUMINUM

No corrosive effects of any type were found in the aluminum specimens. The specimens retained their original metallic luster, Fig. 26, and microscopic examination revealed no microstructural damage, Fig. 27. A control specimen is pictured in Fig. 28.

6.4 STAINLESS STEEL CRYOPANEL SECTION

No indication of corrosive attack, general or localized, was found in this specimen. There was no visible surface deposit. The weld regions were closely examined and there was no evidence of damage. A cross-sectional photomicrograph is shown in Fig. 29. The slightly roughened surface is a characteristic of the metal and was not caused by corrosive attack. A control specimen is shown in Fig. 30.

SECTION VII SUMMARY AND CONCLUSIONS

The following summarization and conclusions are based on the tests reported herein.

1. The exposure conditions used in this test (Section 4.2) did not cause significant microstructural corrosion in any of the cryopanel materials tested.
2. The copper surfaces were tarnished from the exposure. The black epoxy paint, a common cryopanel coating, did not protect the metal surface from tarnishing. The deposit appears to be related to a decomposition of the MMH rather than a reaction product between copper and the propellant. No microstructural damage was found beneath the deposit, but the optical properties of the copper surface were markedly changed.
3. The black epoxy coating was separated from the copper surfaces and appeared to have blistered during the test. Close examination showed that the coating had separated to a large extent from the stainless steel surfaces.
4. No stress-corrosion cracking was detectable in welded specimens exposed with an applied bending stress.
5. All of the metallic materials tested appear to be adequate candidates for use under conditions similar to those used in this test. The change in optical properties of copper surfaces may, however, be significant for certain applications.

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4. Cloyd, D. R. and Murphy, W. J. "Handling Hazardous Materials." NASA SP-5032, September 1965.

**APPENDIX
ILLUSTRATIONS**

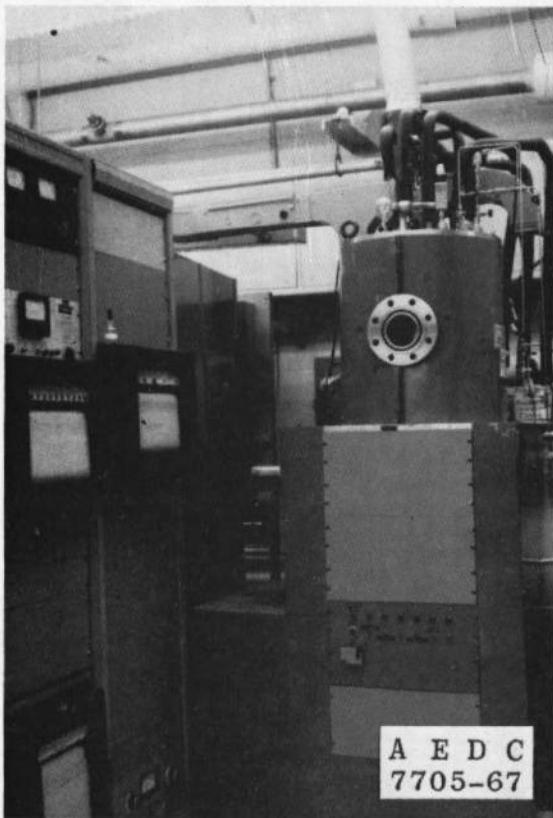


Fig. 1 1.5- by 2-ft Research Vacuum Chamber

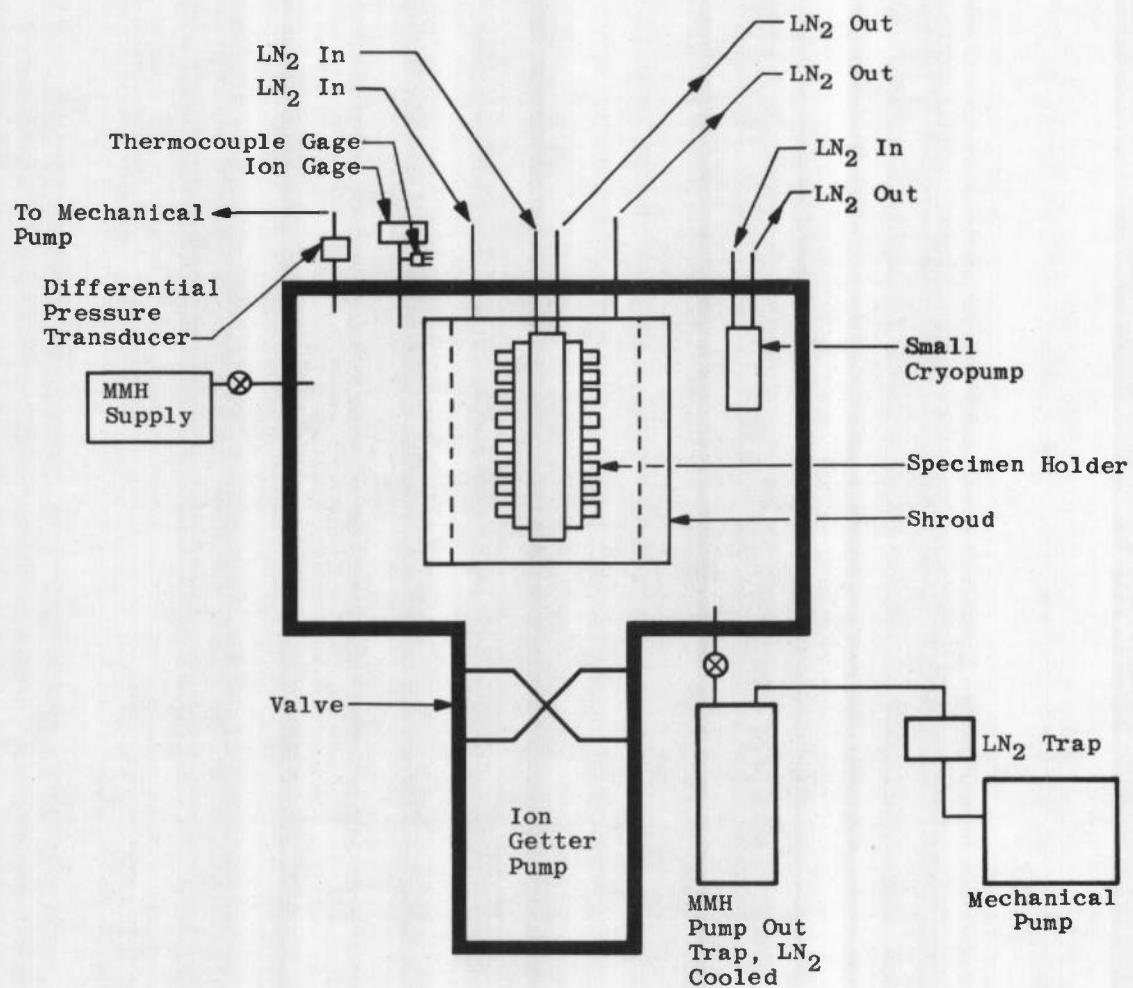


Fig. 2 Schematic, MMH Corrosion Test Chamber

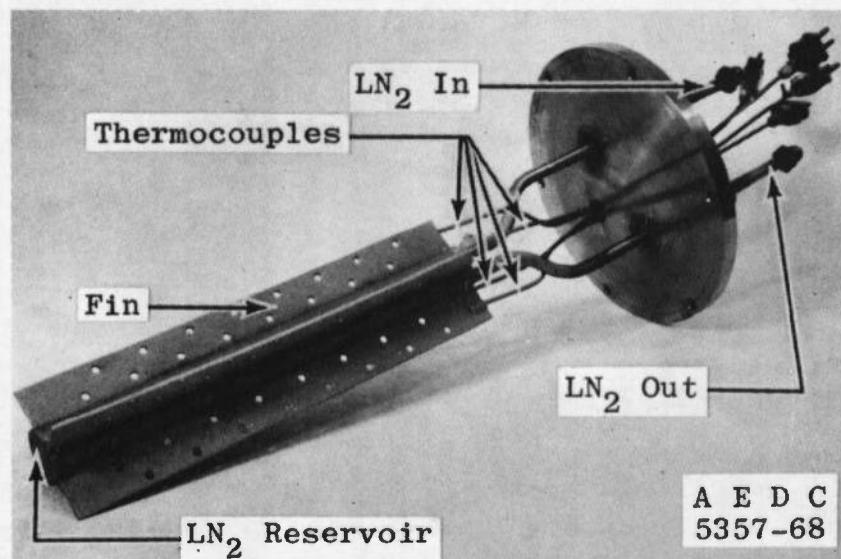


Fig. 3 Specimen Holder for MMH Test

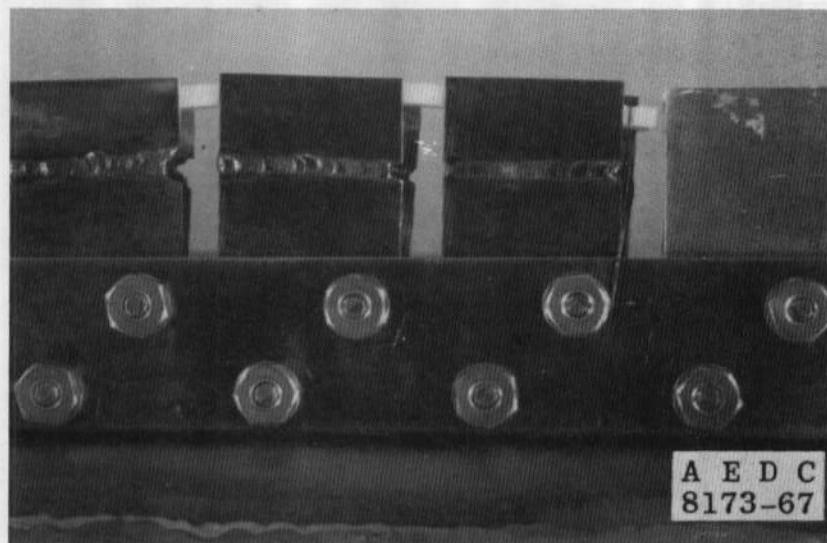


Fig. 4 Test Specimens Mounted in Holder

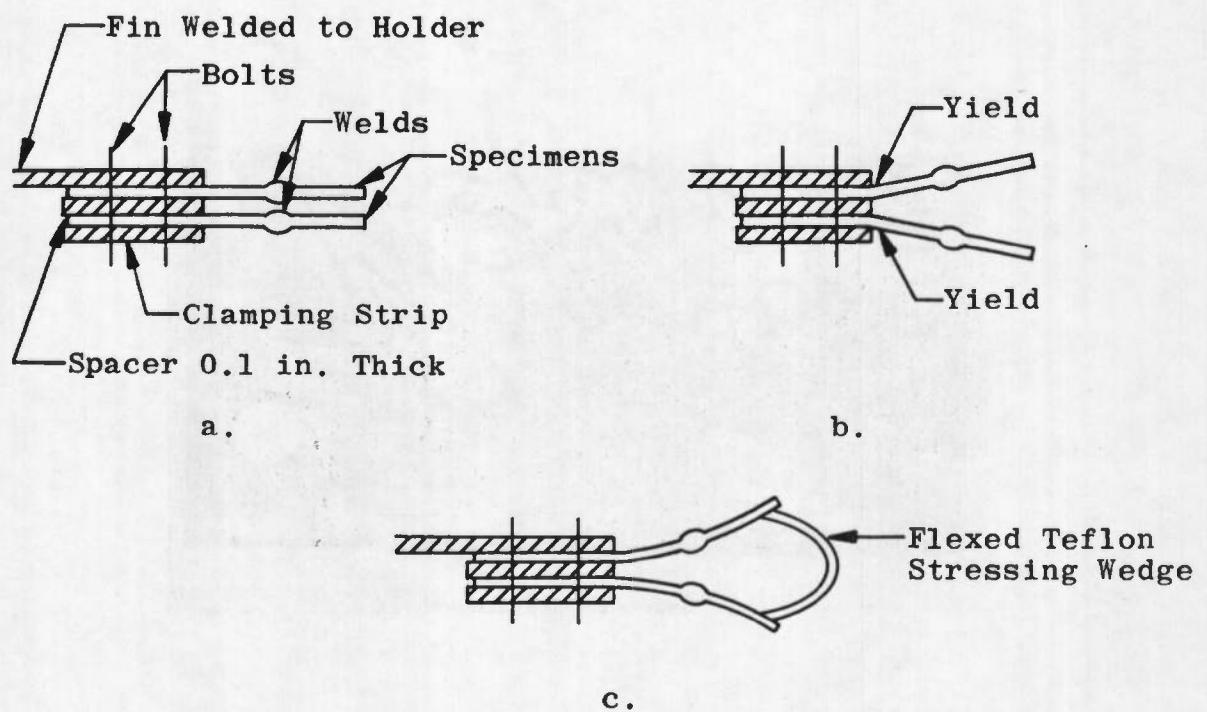


Fig. 5 Method of Stressing Test Specimens

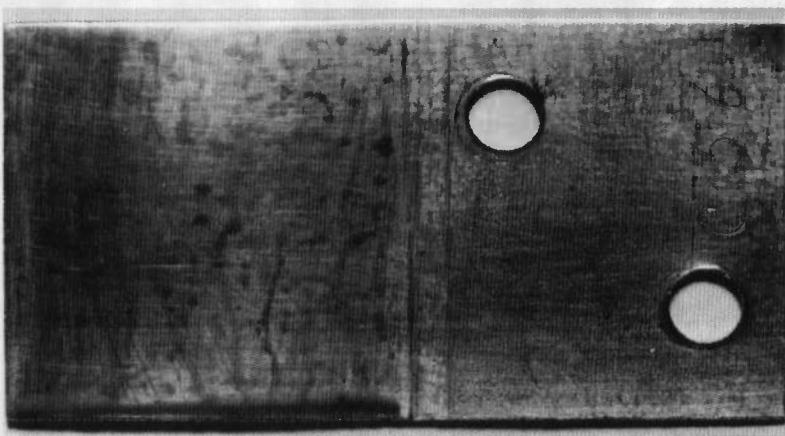


Fig. 6 Copper Exposed to MMH, 2X

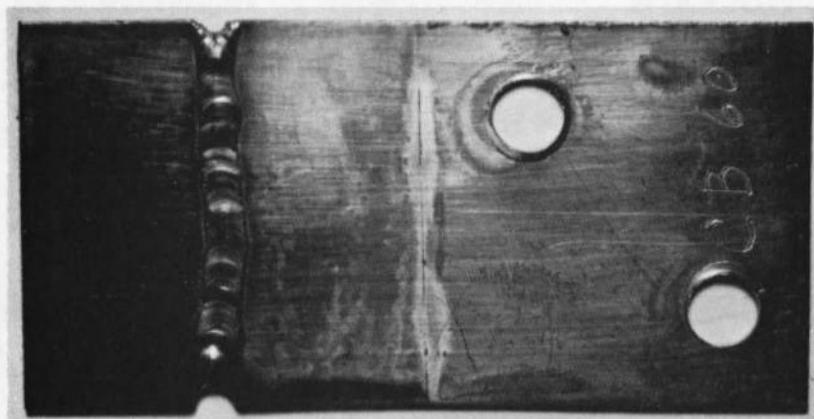


Fig. 7 Brazed Copper Exposed to MMH, 2X

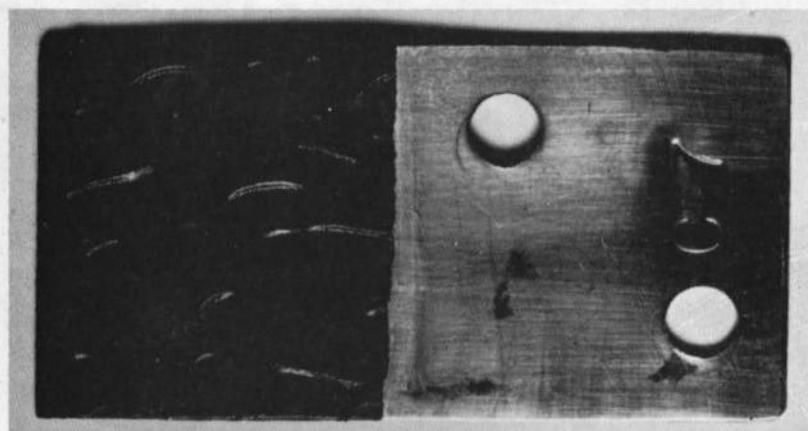


Fig. 8 Coated Copper Exposed to MMH, 2X

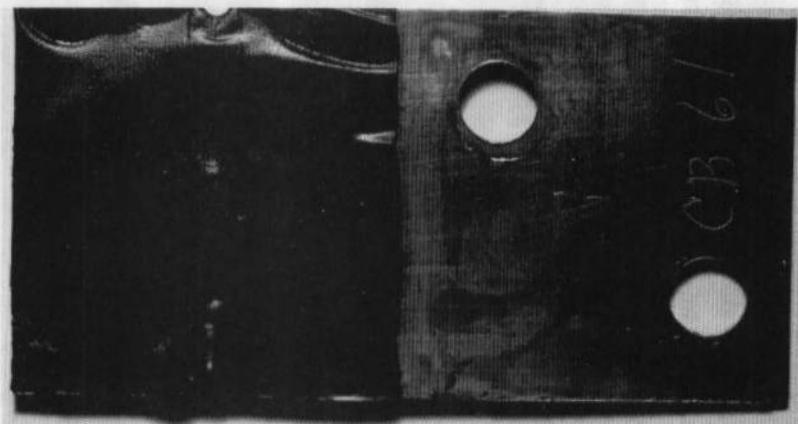


Fig. 9 Coated Brazed Copper Exposed to MMH, 2X



Fig. 10 Coating Stripped from Copper after MMH Exposure, 2X



Fig. 11 Copper Control, Unexposed, 500X

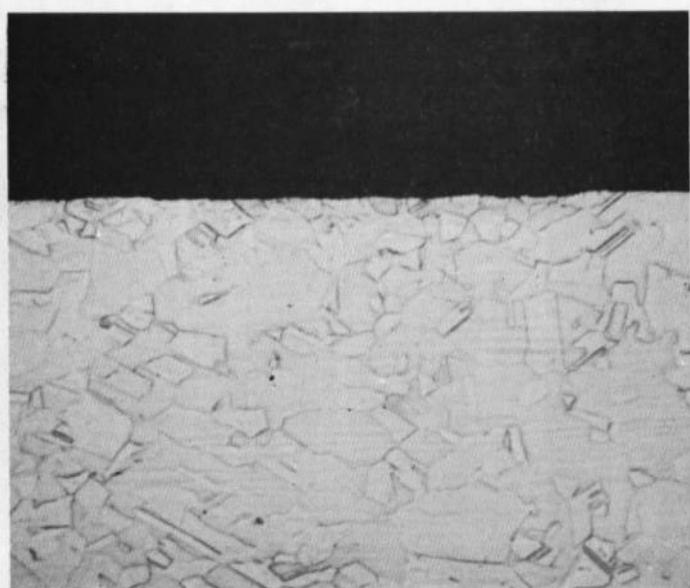


Fig. 12 Uncoated Copper Exposed to MMH, 500X



Fig. 13 Coated Copper, Exposed to MMH, 500X



Fig. 14 Brazed Copper Exposed to MMH, Stressed, and Uncoated,
500X

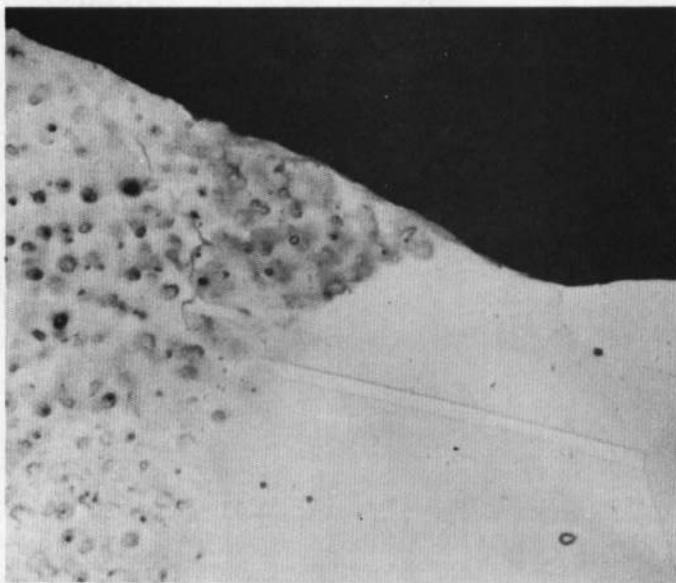


Fig. 15 Control Specimen, Braze Copper (at Weld), Unexposed, and Unstressed, 500X

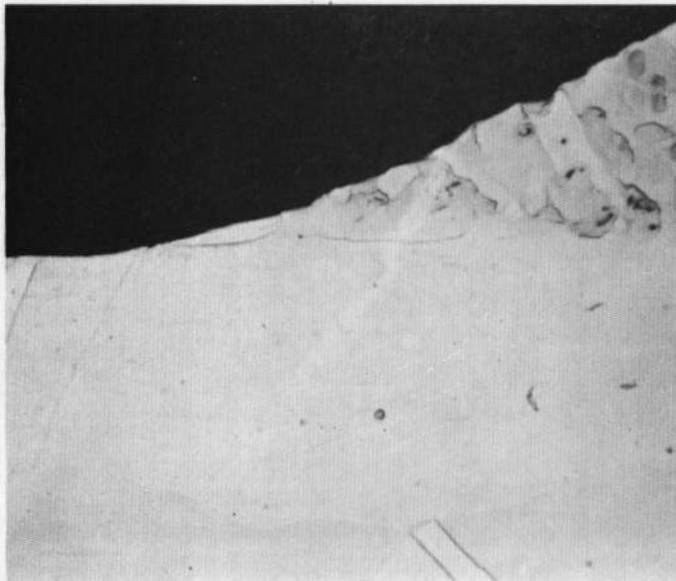


Fig. 16 Braze Copper Exposed to MMH, Stressed (At Weld), 500X

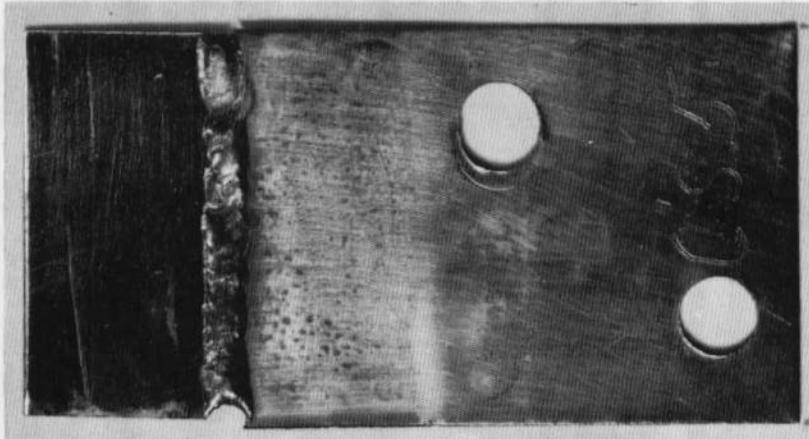


Fig. 17 Composite Exposed to MMH, Uncoated, 2X

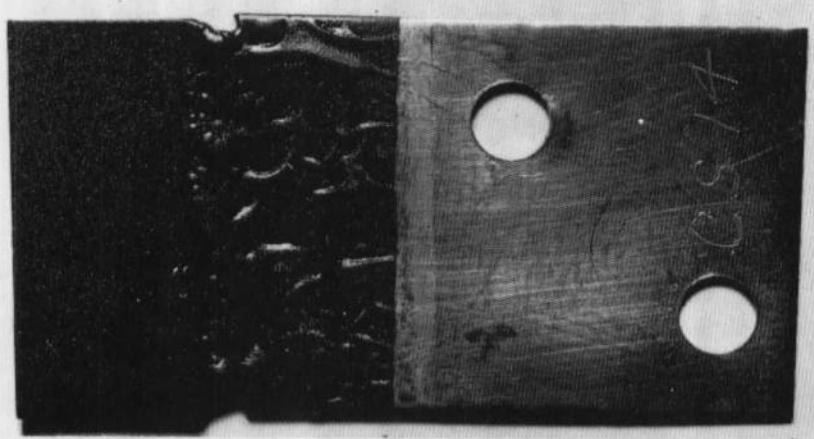


Fig. 18 Composite Coated and Exposed to MMH, 2X

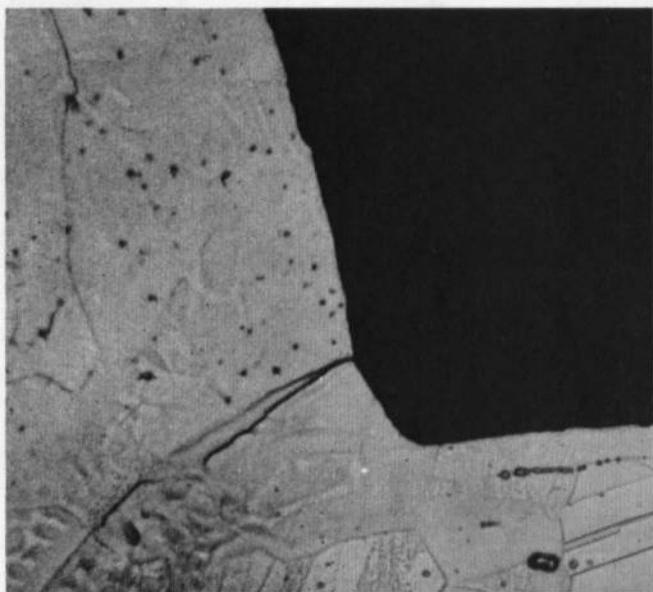


Fig. 19 Composite, Stainless Steel Side of Weld, Stressed and Exposed to MMH, 500X

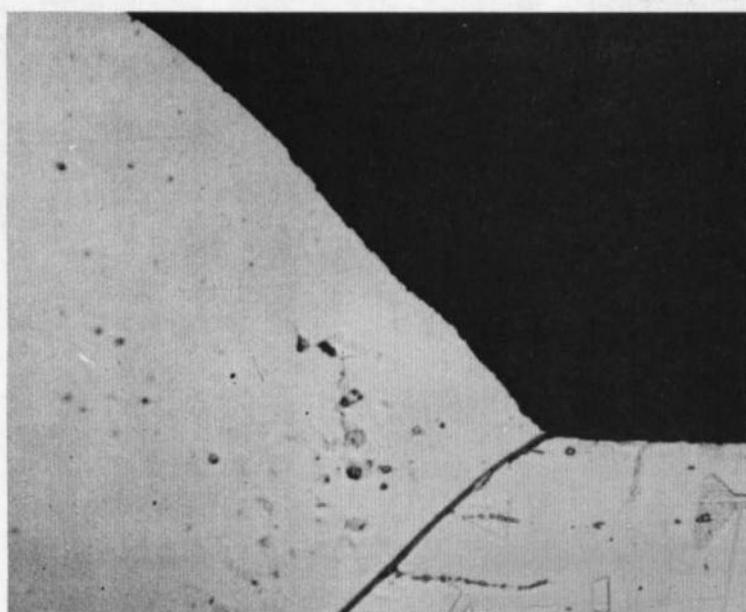


Fig. 20 Control Composite, Stainless Steel Side of Weld, Unexposed, 500X

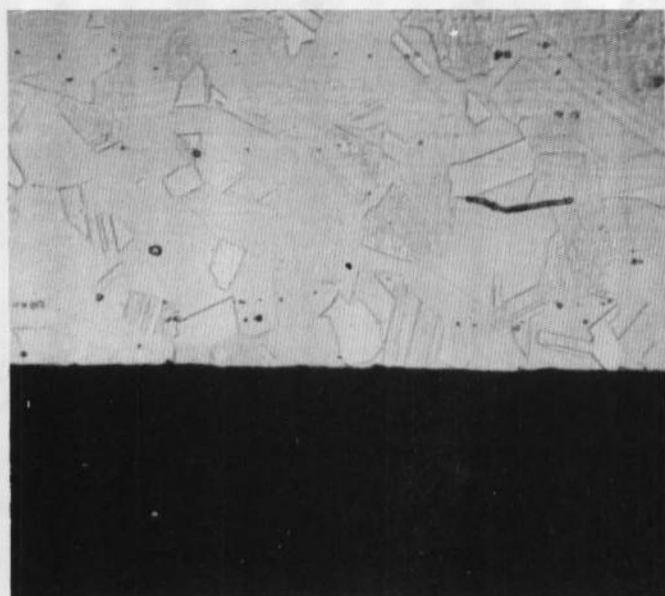


Fig. 21 Composite, Stainless Steel Portion away from Weld, Stressed and Exposed to MMH, 500X

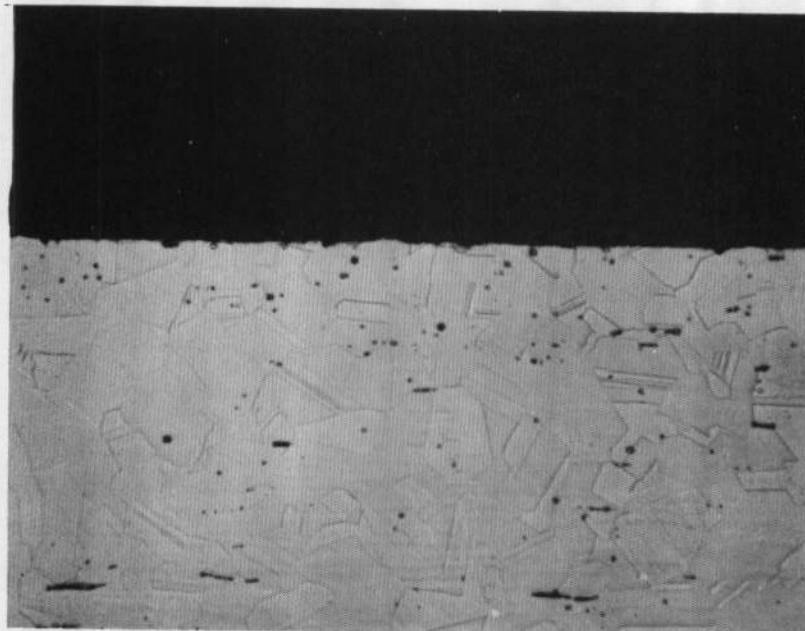


Fig. 22 Control Composite, Unexposed, Stainless Steel Portion, 500X

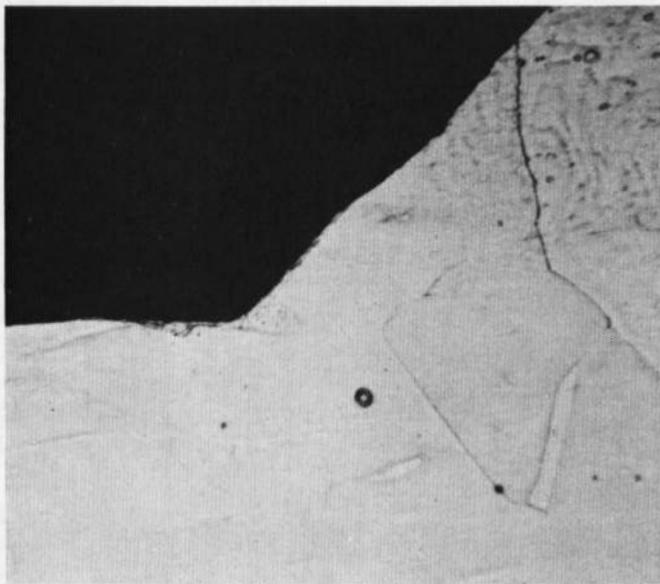


Fig. 23 Composite, Copper Side of Weld, Stressed and Exposed to MMH, 500X

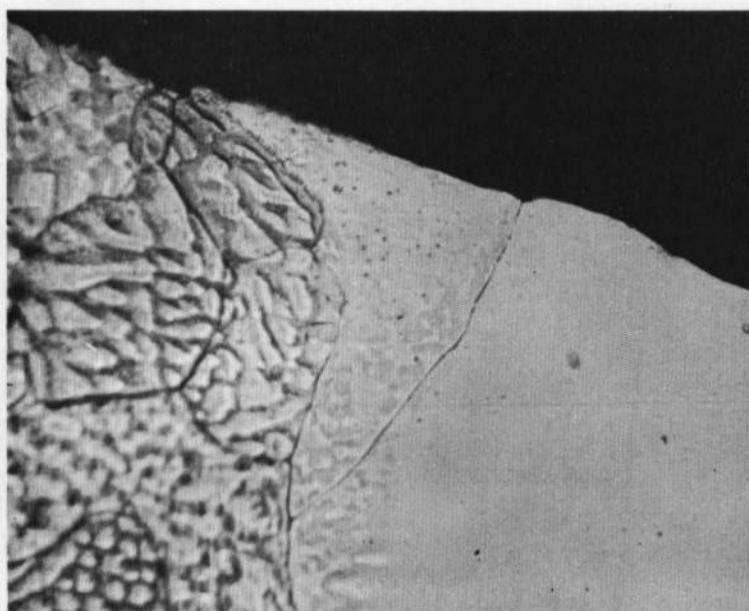


Fig. 24 Control Composite, Copper Side of Weld, Unexposed, 500X



Fig. 25 Composite, Copper Portion away from Weld, Stressed and Exposed to MMH, 500X

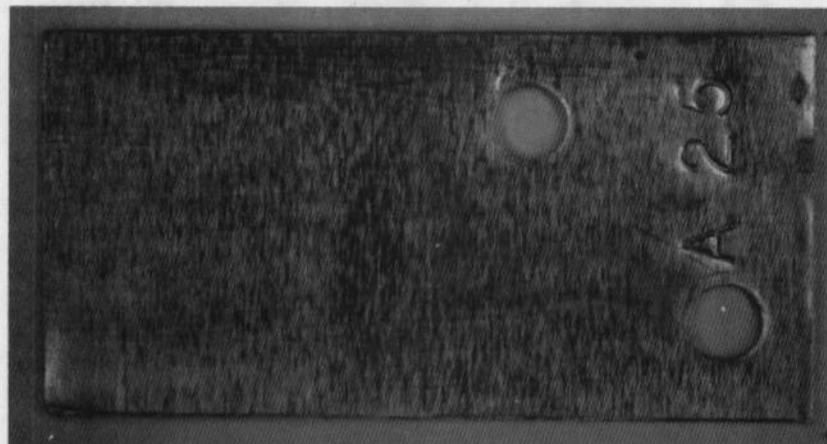


Fig. 26 Aluminum Exposed to MMH, 2X



Fig. 27 Aluminum Exposed to MMH, 500X

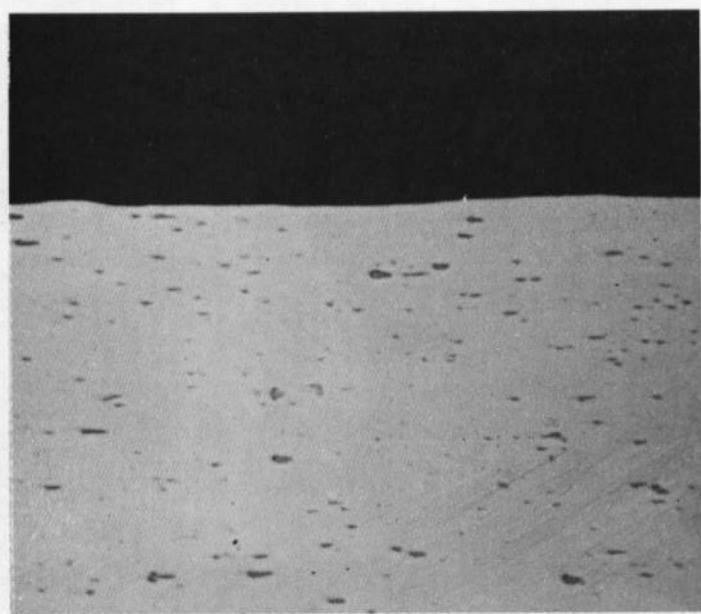


Fig. 28 Control Aluminum, Unexposed, 500X

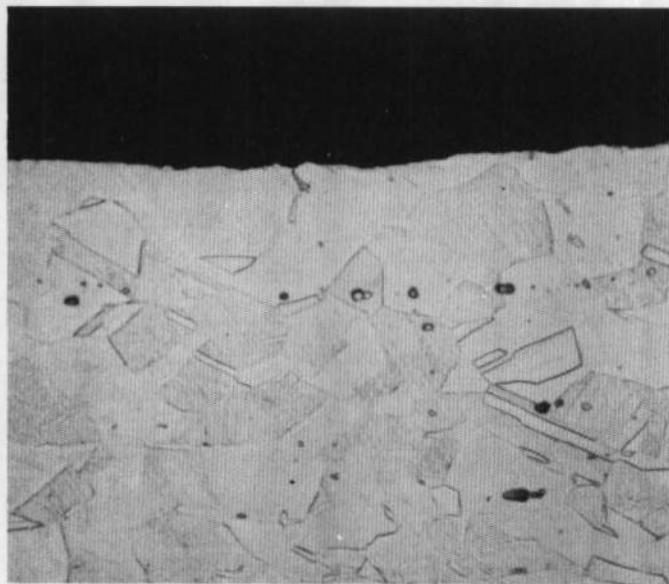


Fig. 29 Stainless Steel Cryopanel Exposed to MMH, 500X



Fig. 30 Control, Stainless Steel Cryopanel, Unexposed, 500X

UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Arnold Engineering Development Center ARO, Inc., Operating Contractor Arnold Air Force Station, Tenn. 37389		2a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED
		2b. GROUP N/A
3. REPORT TITLE EFFECTS OF MONOMETHYLHYDRAZINE ON CRYOPANEL MATERIALS FOR SPACE SIMULATION CHAMBER PROPULSION TESTS		
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Final Report March to August, 1967		
5. AUTHOR(S) (First name, middle initial, last name) P. G. Waldrep and D. M. Trayer, ARO, Inc.		
6. REPORT DATE December 1968	7a. TOTAL NO. OF PAGES 39	7b. NO. OF REFS 4
8a. CONTRACT OR GRANT NO. F40600-69-C-0001	9a. ORIGINATOR'S REPORT NUMBER(S) AEDC-TR-68-194	
b. PROJECT NO. 5730	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) N/A	
c. Task 04		
d. Program Element 62302F		
10. DISTRIBUTION STATEMENT This document has been approved for public release and sale; its distribution is unlimited.		
11. SUPPLEMENTARY NOTES Available in DDC.	12. SPONSORING MILITARY ACTIVITY Arnold Engineering Development Center, Air Force Systems Command, Arnold AF Station, Tenn. 37389	
13. ABSTRACT <p>The effects of monomethylhydrazine on several commonly used cryopanel materials were investigated. The test conditions approximated those expected during rocket engine tests in a space simulation chamber. Monomethylhydrazine gas pressure was 4 torr at room temperature. Test specimen temperatures were varied between 77 and about 300°K. All specimens were tested simultaneously for a total exposure time of 532 hr. Phosphorus deoxidized copper, type 304L stainless steel, and series 1100 aluminum were tested. Some specimens were brazed or welded and some were coated with a black epoxy paint. Several welded and brazed specimens were tested with an imposed bending stress. No microstructural damage occurred to any of the metals. The copper, however, tarnished noticeably, and the cryopanel paint separated from the coated specimens.</p>		

UNCLASSIFIED

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
cryopanels						
monomethylhydrazine						
rocket engines						
propellants						
space simulation chambers						
facility development						
research facilities, aerospace						

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